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FILE 'CAPLUS' ENTERED AT 17:38:09 ON 25 JUN 2003

L1 9 S MALDI (S) "CONTACT LENS"  
L2 0 S MALDI AND (ABSORBENT? (S) ELUANT?)  
L3 0 S MALDI AND (ADSORBENT? (S) ELUANT?)  
L4 0 S MALDI AND ADSORBENT? AND ELUENT?  
L5 0 S MALDI AND (COMBINATOR? OR "HIGH THROUGHPUT" OR MULTI?  
OR PLUR  
L6 765 S MALDI AND (COMBINATOR? OR "HIGH THROUGHPUT" OR MULTI?  
OR PLUR  
L7 75 S L6 AND CONDITION?  
L8 3 S L7 AND (WASH? OR ELUT? OR RINS?)  
L9 164 S MALDI (S) CONDITION?  
L10 9 S L9 AND (ELUANT? OR WASH? OR ADSORB? OR ABSORB?)

L1 ANSWER 1 OF 9 CAPLUS COPYRIGHT 2003 ACS

ACCESSION NUMBER: 2003:185878 CAPLUS

TITLE: "ESI and MALDI-TOF MS analysis of implantable device polymers and their interactions with proteins"

AUTHOR(S): *Maziarz, E. Peter, III; Liu, X. Micheal; Grobe, George L.; Baker, Gary A.; Bonafini, James*

CORPORATE SOURCE: Bausch and Lomb, Rochester, NY, 14603, USA

SOURCE: **Abstracts of Papers, 225th ACS National Meeting, New Orleans, LA, United States, March 23-27, 2003 (2003), PMSE-080. American Chemical Society: Washington, D.C.**

CODEN: 69DSA4

DOCUMENT TYPE: Conference; Meeting Abstract

LANGUAGE: English

AB Implantable device products such as contact lenses, intraocular lenses, and drug delivery devices are made from polymeric materials. Development of these products is enhanced from a more detailed understanding of the oligomer components within the product and their interaction with bodily fluid. The prerequisite to approach such an understanding involves anal. methods that rapidly and unambiguously identify the relevant components in complex mixts. This study is two-fold. First, we demonstrate the unique potential ESI and MALDI-TOF MS offers toward synthetic polymer anal. These studies include hyphenation of GPC with ESI and MALDI-TOF MS to evaluate polydisperse polymers. We obtain detailed information including repeat unit sequence, existence of impurities, and end group chem. of selected polymeric materials. The second part of this report demonstrates the potential for MS to evaluate the interactions between protein and polymer materials. These studies include the anal. of proteins directly from a contact lens using MALDI-TOF MS.

L1 ANSWER 2 OF 9 CAPLUS COPYRIGHT 2003 ACS

ACCESSION NUMBER: 2002:776963 CAPLUS

TITLE: "Analysis of implantable device polymers and their interactions with proteins"

AUTHOR(S): *Maziarz, E. Peter, III; Liu, X. Micheal; Mosack, Linda; Witham, Paula; Grobe, George L.*

CORPORATE SOURCE: Bausch and Lomb, Rochester, NY, 14603, USA

SOURCE: **Abstracts of Papers, 224th ACS National Meeting, Boston, MA, United States, August 18-22, 2002 (2002), PMSE-232. American Chemical Society: Washington, D.C.**

CODEN: 69CZPZ

DOCUMENT TYPE: Conference; Meeting Abstract

LANGUAGE: English

AB Development of a product, such as an implantable device, is enhanced from a more complete understanding of the underlying principles that govern the mol. components within the product and their interaction with bodily fluid. The prerequisite to approach such an understanding involves anal. methods that rapidly and unambiguously identify all relevant components in complex mixts. By merit of its high sensitivity, specificity, selectivity, and information-rich nature, matrix assisted laser desorption ionization (MALDI) and electrospray ionization (ESI) time of flight (TOF) mass spectrometry (MS) can be an indispensable anal. tool for characterization of protein and polymeric materials. The crux of this study is two-fold. First, we demonstrate the unique potential ESI and MALDI-TOF MS offers toward synthetic polymer anal. These studies include hyphenation of gel permeation chromatog. (GPC) with ESI and MALDI-TOF MS to evaluate polydisperse polymers and complex mixts. thereof. We obtain detailed information including repeat unit sequence, existence of impurities, and end group chem. of selected polymeric materials. The second part of this report demonstrates the potential for mass spectrometry to evaluate the interactions between protein and polymer materials. These studies include the anal. of proteins directly from a contact lens using MALDI-TOF MS. Also the unfolding of lysozyme protein in the presence of select polymer materials is demonstrated through hydrogen/deuterium exchange expts. Though only preliminary steps toward a more complete understanding of contact lens materials, this report provides an overview of MALDI and ESI-TOF MS and demonstrates the ability of these techniques to evaluate polymer materials and probe for polymer-protein interactions.

L1 ANSWER 3 OF 9 CAPLUS COPYRIGHT 2003 ACS

ACCESSION NUMBER: 2002:185638 CAPLUS

DOCUMENT NUMBER: 136:213183

TITLE: Quantitative MALDI-time of flight mass spectrometry of peptides and proteins

INVENTOR(S): **Ammon, Daniel M.**

PATENT ASSIGNEE(S): USA

SOURCE: U.S. Pat. Appl. Publ., 45 pp.

CODEN: USXXCO

DOCUMENT TYPE: Patent

LANGUAGE: English

PATENT NO. KIND DATE APPLICATION NO. DATE

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US 2002031773 A1 20020314 US 2001-876412 20010607

PRIORITY APPLN. INFO.: US 2000-210074P P 20000607

AB A method of quant. analyzing a sample analyte involves performing matrix-assisted laser desorption ionization mass spectrometry on the sample analyte and an internal std., and comparing the mass spectrometry of the sample analyte with the mass spectrometry of the internal std.

L1 ANSWER 4 OF 9 CAPLUS COPYRIGHT 2003 ACS

ACCESSION NUMBER: 2001:759022 CAPLUS

DOCUMENT NUMBER: 137:68116

TITLE: "XPS and surface-MALDI-MS characterization of worn HEMA-based contact lenses"

AUTHOR(S): *McArthur, S. L.; McLean, K. M.; St. John, H. A. W.; Griesser, H. J.*

CORPORATE SOURCE: Cooperative Research Centre for Eye Research and Technology, CSIRO Molecular Science, Clayton, 3169, Australia

SOURCE: **Biomaterials (2001), 22(24), 3295-3304**

CODEN: BIMADU; ISSN: 0142-9612

PUBLISHER: Elsevier Science Ltd.

DOCUMENT TYPE: Journal

LANGUAGE: English

AB XPS and MALDI-MS were used to analyze initial adsorption events in the fouling of HEMA-based contact lenses. All of the lenses tested accumulated tear film deposits within 10 min of wear. XPS indicated the presence of mainly proteinaceous deposits, with indications of some contributions by mucins or lipids on some lenses and the nature of the deposit being influenced by the lens chem. MALDI-MS detected the presence of surface-adsorbed species with mol. wts. <15 kDa. While lysozyme could be identified by comparison of MALDI-MS signals with known protein mass and assignments are suggested for some other signals, several other species, with MWs less than that of lysozyme, could not be identified as no ocular proteins with corresponding MWs had been reported in previous biochem. tear film analyses. These species, and others, were also detected in MALDI-MS anal. of reflex tear film, suggesting that the adsorbed unidentified species were not simply products of surface-induced dissocn. of adsorbing higher-MW proteins. This short-term wear study detected rapid interface conversion and demonstrated the utility and surface sensitivity of XPS and MALDI-MS in characterizing contact lens deposits at the initial stages when sub-monolayer adsorbed amts. are present on lenses.

REFERENCE COUNT: 36

L1 ANSWER 5 OF 9 CAPLUS COPYRIGHT 2003 ACS

ACCESSION NUMBER: 1999:780099 CAPLUS

DOCUMENT NUMBER: 132:113021

TITLE: "Matrix-assisted laser desorption ionization mass spectrometry detection of proteins adsorbed in vivo onto contact lenses"

AUTHOR(S): *Kingshott, Peter; St John, Heather A. W.; Chatelier, Ronald C.; Griesser, Hans J.*

CORPORATE SOURCE: CSIRO Molecular Science, Clayton, 3169, Australia

SOURCE: **Journal of Biomedical Materials Research (2000), 49(1), 36-42**

R 857. M3

B 568

CODEN: JBMRBG; ISSN: 0021-9304

PUBLISHER: John Wiley & Sons, Inc.

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Identification of the biomols. that form the first adsorbed monolayer, which thus effect "interface conversion", in competitive adsorption from multicomponent biol. solns. can be challenging because of limitations in mass resoln. and sensitivity of established techniques. In this study matrix-assisted laser desorption ionization (MALDI) time of flight mass spectrometry is developed and applied as a novel surface anal. method to enable anal. of adsorbed multicomponent biomol. layers directly on the biomaterial surfaces. We show that proteins adsorbed in vivo (on human eyes) on contact lenses can be detected rapidly and conveniently by the diagnostic highly resolved mass signals recorded by MALDI mass spectrometry. This new approach allows detection of minor (and major) proteinaceous constituents of biofouled layers at levels substantially below monolayer coverage. Identification is done by comparison with mol. masses of known proteins. Specifically, it is shown that in addn. to lysozyme, other low mol. wt. proteins adsorb from human tear fluid onto contact lenses; these proteins had not been detected in earlier studies using other techniques.

REFERENCE COUNT: 17

L1 ANSWER 6 OF 9 CAPLUS COPYRIGHT 2003 ACS

ACCESSION NUMBER: 1999:558143 CAPLUS

DOCUMENT NUMBER: 131:319764

TITLE: "Direct Detection of Proteins Adsorbed on Synthetic Materials by Matrix-Assisted Laser Desorption Ionization-Mass Spectrometry"

AUTHOR(S): *Kingshott, Peter; St. John, Heather A. W.; Griesser, Hans J.*

CORPORATE SOURCE: CSIRO Molecular Science, Clayton South MDC, 3169, Australia

SOURCE: **Analytical Biochemistry (1999), 273(2), 156-162**

CODEN: ANBCA2; ISSN: 0003-2697

PUBLISHER: Academic Press

DOCUMENT TYPE: Journal

LANGUAGE: English

AB The irreversible accumulation of biol. material on synthetic surfaces ("biofouling") adversely affects for instance contact lenses, implantable biomedical devices, biosensors, water purifn., transport and storage systems, and marine structures. It is shown here that proteins adsorbed on contact lenses can be detected directly, rapidly, and conveniently, with high sensitivity, by matrix-assisted laser desorption ionization (MALDI)-mass spectrometry. This new approach allows detection of minor (and major) proteinaceous constituents of biofouled layers on samples retrieved from clin. usage and in vitro protein adsorption studies, at levels substantially below monolayer coverage. Identification of the detected biol. mols. can be done by comparison of the detected mass peaks with known protein mol. masses or with spectra recorded of pure compds. or by sep. biochem. assays. The MALDI mass spectra recorded on different contact lenses contain peaks assignable to lysozyme and a no. of smaller proteins. Such sensitive characterization of the early stages of biofouling enhances the understanding of protein/materials interactions

and assists in designing guided strategies toward control of biol. adsorption processes.

(c) 1999 Academic Press.

REFERENCE COUNT: 23

L1 ANSWER 7 OF 9 CAPLUS COPYRIGHT 2003 ACS

ACCESSION NUMBER: 1997:252294 CAPLUS

DOCUMENT NUMBER: 126:282741

TITLE: "Study of protein adsorption onto polysaccharide contact lens coatings by MALDI-TOF-MS and XPS"

AUTHOR(S): *Kingshott, Peter; St. John, Heather A. W.; Chatelier, Ronald C.; Griesser, Hans J.*

CORPORATE SOURCE: Division of Chemicals and Polymers, CSIRO, Clayton South MDC, Clayton, 3169, Australia

SOURCE: **Polymeric Materials Science and Engineering (1997), 76, 81-82**

CODEN: PMSE DG; ISSN: 0743-0515

PUBLISHER: American Chemical Society

DOCUMENT TYPE: Journal

LANGUAGE: English

AB The combination of XPS and MALDI-TOF-MS is suitable for the study of protein adsorption to contact lens coating. It enables quantification of adsorbed protein amts. and identification of the major species present, by the unique ability of MALDI to furnish mol. ion signals that unambiguously identify the proteins.

TP156.C57

A5

L1 ANSWER 8 OF 9 CAPLUS COPYRIGHT 2003 ACS

ACCESSION NUMBER: 1997:163609 CAPLUS

TITLE: "Surface characterization of worn hydrogel contact lenses".

AUTHOR(S): *St John, H. A. W.; Kingshott, P.; Griesser, H. J.; Morris, C.; Bolis, S.*

CORPORATE SOURCE: Division Chemicals and Polymers, CSIRO, Clayton, 3169, Australia

SOURCE: Book of Abstracts, 213th ACS National Meeting, San Francisco, April 13-17 (1997), PMSE-049. American Chemical Society: Washington, D. C.

CODEN: 64AOAA

DOCUMENT TYPE: Conference; Meeting Abstract

LANGUAGE: English

AB Surface sensitive techniques such as XPS, SSIMS and MALDI-MS have been used to study the initial stages of interface conversion for hydrogel contact lenses worn by human patients. Proteinaceous material was found to be irreversibly bound to lens surfaces within 5 s of wear. The rate of further deposition was shown to dependent on the patient and compn. of the lens polymer. The spatial distribution of deposits on the front and back surfaces of the lens was also dependent on the polymer compn. Lipid deposition was highly patient dependent and independent of protein adsorption. SIMS anal. confirmed the conversion of the outermost surface of Acuvue lenses to mostly proteinaceous material within 4 h of wear. MALDI-MS identified the deposition of lysozyme on all worn hydrogel lenses, and also lower mol. wt. species such as protein fragments.

L1 ANSWER 9 OF 9 CAPLUS COPYRIGHT 2003 ACS

ACCESSION NUMBER: 1997:163608 CAPLUS

TITLE: "Study of protein adsorption onto polysaccharide contact lens coatings by MALDI-TOF-MS and XPS".

AUTHOR(S): *Kingshott, P.; St John, H. A. W.; Chatelier, R. C.; Griesser, H. J.*

CORPORATE SOURCE: Division Chemicals and Polymers, CSIRO, Clayton, 3169, Australia

SOURCE: **Book of Abstracts, 213th ACS National Meeting, San Francisco, April 13-17 (1997), PMSE-048. American Chemical Society: Washington, D. C.**

CODEN: 64AOAA

DOCUMENT TYPE: Conference; Meeting Abstract

LANGUAGE: English

AB We utilise matrix-assisted laser desorption ionisation time-of-flight mass spectrometry (MALDI-TOF-MS) and XPS (XPS) to study the interfacial interactions between tear proteins, from an artificial tear fluid formulation (ATF), and polysaccharide (PS) contact lens coatings. All PS coatings exhibited excellent water wettability and their and XPS confirmed that a uniform PS coating is successfully attached to the aminated n-heptylamine plasma polymer film using our attachment chem. XPS also shows that all PS surfaces have a strong affinity for the proteins of ATF but the rate of protein adsorption varies between PS surfaces. MALDI-TOF-MS directly identifies which species of protein exists on each surface by the generation of intact protein mol. ions. For example, our oxidised dextran (OD) surface contains both adsorbed lysozyme and lactoferrin after immersion in ATF for 1 h.

L8 ANSWER 1 OF 3 CAPLUS COPYRIGHT 2003 ACS

ACCESSION NUMBER: 2001:817049 CAPLUS

DOCUMENT NUMBER: 135:341206

TITLE: A quantitative, high-throughput screening method for protein stability

INVENTOR(S): Oas, Terrance G.; Ghaemmaghami, Sina; Powell, Kendall D.; Fitzgerald, Michael C.

PATENT ASSIGNEE(S): Duke University, USA

SOURCE: PCT Int. Appl., 160 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent

LANGUAGE: English

PATENT NO. KIND DATE APPLICATION NO. DATE

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WO 2001084137 A1 20011108 WO 2001-US13907 20010430

EP 1307736 A1 20030507 EP 2001-928981 20010430

PRIORITY APPLN. INFO.: US 2000-200311P P 20000428

WO 2001-US13907 W 20010430

AB The invention concerns the screening of a large no. of polypeptides for the presence of stable structures. Described, herein, are methods [referred to as MALDI MS-HX and SUPREX (stability of unpurified proteins from rates of H/D exchange)] for measuring the stability of proteins in a rapid high-throughput fashion. The method employs hydrogen exchange with deuterium to est. the stability of quantities of unpurified protein exts.,

using matrix-assisted laser desorption/ionization (MALDI) mass spectrometry. The method includes the steps of (a) providing a test protein; (b) contacting the protein with an exchange buffer comprising a denaturant and deuterium, the exchange buffer having a denaturant concn.; (c) contacting the protein with a mass spectrometry matrix medium; (d) detg. a change in mass of the test protein by mass spectrometry; (e) varying the denaturant concn. of the exchange buffer; (f) repeating steps (a)-(e) a desired no. of times; and (g) quant. detg. protein stability based on the change in mass of the test protein as a function of denaturant concn., whereby the stability of a test protein under the native conditions is quant. detd. The stabilities of maltose binding protein and monomeric  $\lambda$  repressor variants detd. by SUPREX agree well with stability data obtained from conventional CD denaturation of purified protein. The method also can detect the change in stability caused by the binding of maltose to maltose binding protein. The results demonstrate the precision of the method over a wide range of stabilities.

REFERENCE COUNT: 9

L8 ANSWER 2 OF 3 CAPLUS COPYRIGHT 2003 ACS

ACCESSION NUMBER: 2000:27374 CAPLUS

DOCUMENT NUMBER: 132:163109

TITLE: "Effect of experimental conditions on the analysis of sodium dodecyl sulphate polyacrylamide gel electrophoresis separated proteins by matrix-assisted laser desorption/ionisation mass spectrometry"

AUTHOR(S): *Galvani, Marina; Bordini, Ellenia; Piubelli, Chiara; Hamdan, Mahmoud*

CORPORATE SOURCE: Glaxo Wellcome Medicines Research Centre, Verona, 37135, Italy

SOURCE: **Rapid Communications in Mass Spectrometry (2000), 14(1), 18-25**

CODEN: RCMSEF; ISSN: 0951-4198

PUBLISHER: John Wiley & Sons Ltd.

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Two mixts. of proteins having mol. wts. in the range .apprx.8-97 kDa were sepd. by SDS-PAGE (SDS-PAGE) and examd. by delayed extn. matrix-assisted laser desorption/ionization mass spectrometry (MALDI-MS). Part of our aim in this study is to gain more insight into the influence of the various exptl. conditions on the overall quality of the acquired mass spectral data. Different protein extn. procedures, two staining agents, and extn. times, were among the parameters assessed. In terms of the overall quality of the acquired mass spectra and the speed of protein recovery, ultrasonic assisted passive elution, into a solvent mixt. contg. formic acid/acetonitrile/2-isopropanol/water, was found to be more efficient than other elution procedures. The higher resoln. assocd. with the delayed extn. mode allowed the identification of a no. of protein modifications, including multiple formylation provoked by formic acid, cysteine alkylation caused by unpolymd. acrylamide monomers, and complexation with the staining reagents. The detection of these modifications, however, was limited to proteins under 30 kDa. Anal. of a ubiquitin tryptic digest by reflectron MALDI time-of-flight (TOF) allowed reliable identification of a no. of the formylation sites.

REFERENCE COUNT: 28

L8 ANSWER 3 OF 3 CAPLUS COPYRIGHT 2003 ACS

ACCESSION NUMBER: 1996:394661 CAPLUS

DOCUMENT NUMBER: 125:109548

TITLE: Molecular anatomy of brain using multi-dimensional HPLC-ESI/MS or MALDI/TOFMS

AUTHOR(S): Isobe, Toshiaki; Nakayama, Hiroshi; Shinkai, Fumiko; Yamaguchi, Mihoko; Kanai, Michiko; Ikezawa, Hidenori; Seta, Kazuo; Okuyama, Tsuneo

CORPORATE SOURCE: Tokyo Metropolitan University, Japan

SOURCE: Kuromatogurafi (1996), 17(2), 160-161

CODEN: KUROE9; ISSN: 0917-3048

PUBLISHER: Kuromatogurafi Kagakkai

DOCUMENT TYPE: Journal

LANGUAGE: Japanese

AB The authors combined a multi-dimensional HPLC system to an ESI/MS or a MALDI/TOF-MS for the mol. anatomy of brain. The multi-dimensional HPLC system used an anion-exchange HPLC with a QAE anion-exchange column (TSK 540-QAE) as the 1st dimension and a tandem reversed phase C8 and C18 HPLC columns as the 2nd and 3rd dimension columns, resp. When 500 .mu.L of brain ext. was injected into the anion-exchange column, a linear gradient elution was carried out using water as the initial eluate and a Tris-HCl buffer (pH 7.5) contg. 1.0M NaCl as the terminal eluate. After a protein peak fraction in the QAE anion-exchange HPLC was transferred into a protein trap reversed-phase column (Superspher RP-8e, LichroCART 4 .times. 25 mm) which was located in a 10-port valve. The protein trap column was rinsed with water and then connected in series with a reversed-phase mini-column (LiChrospher 300 RP-8, 4 .times. 4 mm) and an anal. reversed-phase HPLC column (Capcell Pak C18, 2 .times. 50 mm). Brain proteins in the trap column located in the 10-port valve were eluted by a gradient elution from 0.1% TFA/water to 0.1% TFA/acetonitrile along with the reversed-phase mini-column and the anal. reversed-phase column. After the 1st run of reversed phase HPLC, subsequent runs were carried out for the next peak fraction of the anion-exchange HPLC using the same multi-dimensional HPLC conditions. By the use of multi-dimensional HPLC-ESI/MS, accurate mol. wts. of the brain proteins were detd. On the other hand, as an off-line multi -dimensional HPLC-MALDI/MS, fractions collected after the reversed-phase HPLC were also applied for MALDI/MS.

L10 ANSWER 2 OF 9 CAPLUS COPYRIGHT 2003 ACS

ACCESSION NUMBER: 2001:338933 CAPLUS

DOCUMENT NUMBER: 135:2448

TITLE: "Two-layer sample preparation method for MALDI mass spectrometric analysis of protein and peptide samples containing sodium dodecyl sulfate"

AUTHOR(S): Zhang, Nan; Doucette, Alan; Li, Liang

CORPORATE SOURCE: Department of Chemistry, University of Alberta, Edmonton, AB, T6G 2G2, Can.

SOURCE: *Analytical Chemistry* (2001), 73(13), 2968-2975

CODEN: ANCHAM; ISSN: 0003-2700

PUBLISHER: American Chemical Society

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Sodium dodecyl sulfate (SDS) is widely used in protein sample workup. However, many mass spectrometric methods cannot tolerate the presence of this strong surfactant in a protein sample. We present a practical and robust technique based on a two-layer matrix/sample deposition method for the anal. of protein and peptide samples contg. SDS by matrix-assisted laser desorption ionization mass spectrometry (MALDI-MS). The two-layer method involves the deposition of a mixt. of sample and matrix on top of a thin layer of matrix crystals. It was found that for SDS-contg. samples, the intensity of the MALDI signals can be affected by the conditions of sample prepn.: on-probe washing, choice of matrix, deposition method, solvent system, and protein-to-SDS ratio. However, we found that, under appropriate conditions, the two-layer method gave reliable MALDI signals for samples with levels of SDS up to .apprx.1%. The applications of this method are demonstrated for MALDI anal. of hydrophobic membrane proteins as well as bacterial exts. We envision that this two-layer method capable of handling impure samples including those contg. SDS will play an important role in protein mol. wt. anal. as well as in proteome identification by MALDI-MS and MS/MS.

REFERENCE COUNT: 34

L10 ANSWER 3 OF 9 CAPLUS COPYRIGHT 2003 ACS

ACCESSION NUMBER: 2001:255640 CAPLUS

DOCUMENT NUMBER: 135:144281

TITLE: Mechanisms of energy deposition in infrared matrix-assisted laser desorption/ionization mass spectrometry

AUTHOR(S): Menzel, C.; Dreisewerd, K.; Berkenkamp, S.; Hillenkamp, F.

CORPORATE SOURCE: Institut für Medizinische Physik und Biophysik, Universität Münster, Münster, D-48149, Germany

SOURCE: International Journal of Mass Spectrometry (2001), 207(1/2), 73-96

CODEN: IMSPF8; ISSN: 1387-3806

PUBLISHER: Elsevier Science B.V.

DOCUMENT TYPE: Journal

LANGUAGE: English

AB The mechanisms of energy deposition in matrix-assisted laser desorption/ionization mass spectrometry with IR lasers (IR-MALDI-MS) were evaluated in expts. In a 1st part, the threshold fluences for the generation of IR-MALDI spectra were detd. between 2.7 and 4.0  $\mu\text{m}$  wavelength with an optical parametric oscillator as a tunable laser source for nine solid state and two liq. matrixes of different chem. structure and compared to the IR absorption spectra of the compds. Preliminary spectra of IR-MALDI in the wavelength range of 1.45-1.75  $\mu\text{m}$  are also presented using the overtone vibrations of a glycerol matrix. Matrixes were chosen with regard to their IR-MALDI performance and to allow conclusions on the IR-absorption mechanisms. Whereas the wavelength dependence of the threshold fluence for nonhydrogen-bound C-H vibrations essentially follows the absorption spectrum of this functional group, strong discrepancies between the spectral dependence of threshold fluences and IR-absorption spectra were found for the vibrations of O-H and N-H groups around 3- $\mu\text{m}$  wavelength that form strong intermol. and intramol. H bonds. In a 2nd part, expts. are described that interrogate the two most probable mechanisms for the obsd. deviation of the threshold fluence behavior

from the wavelength course of the IR-absorption spectra, i.e., absorption by either free or weakened O-H and N-H stretching modes or by residual free H<sub>2</sub>O. All studies were performed with glycerol and succinic acid as examples for common liq. and solid state matrixes for IR-MALDI. For glycerol, a fluence-dependent, dynamic change in absorption during the laser pulse was revealed by laser transmission measurements on thin glycerol layers. This effect, characterized by a significant blue shift of the O-H stretch absorption, can be attributed to a weakening of intermol. H bonds caused by the transient laser heating of the sample. Taking this effect into account, a good correspondence of the wavelength dependence of the threshold fluence with the IR absorption under IR- MALDI conditions is derived for glycerol. For succinic acid, in contrast, the identification of the predominant absorption mechanism in the 3- $\mu$ m wavelength range appears more difficult. A fluence-dependent absorption was not detected in laser transmission expts. with succinic acid single crystals. A change in analyte-to-matrix ratio, with the intention of inducing free absorbers near crystal defects, also did not influence the wavelength dependence of the threshold fluences. However, an influence of the surface-to-vol. ratio on the wavelength-dependent threshold fluences was found by a comparison of three different prepn. techniques for succinic acid, indicating a putative influence of weakly H-bound surface absorbers. In combination with the detailed anal. of the wavelength dependence of the threshold fluence given from the 1st part, a detn. of the IR-MALDI process for succinic acid based on the absorption by weakly H-bound hydroxyl groups is suggested. No evidence for a significant contribution of residual free H<sub>2</sub>O absorption to the low-threshold fluences around 3- $\mu$ m wavelength was found by monitoring a possible change in threshold fluence at the phase transition from H<sub>2</sub>O to ice and by reducing the analyte hydration and varying the H<sub>2</sub>O content in glycerol preps. Also, in preps. with frozen hydrated proteins without org. matrixes, the wavelength dependence of the threshold fluence did not reflect the spectral absorption of ice, supporting the assumption of a rather minor role of the absorption by residual H<sub>2</sub>O in IR-MALDI.

REFERENCE COUNT: 57

L10 ANSWER 4 OF 9 CAPLUS COPYRIGHT 2003 ACS

ACCESSION NUMBER: 2000:120312 CAPLUS

DOCUMENT NUMBER: 132:331517

TITLE: "A Method for Application of Samples to Matrix-Assisted Laser Desorption Ionization Time-of-Flight Targets That Enhances Peptide Detection"

AUTHOR(S): Landry, France; Lombardo, Christian R.; Smith, Jeffrey W.

CORPORATE SOURCE: Program on Cell Adhesion, The Burnham Institute, La Jolla, CA, 92037, USA

SOURCE: **Analytical Biochemistry (2000), 279(1), 1-8**

CODEN: ANBCA2; ISSN: 0003-2697

PUBLISHER: Academic Press

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Matrix-assisted laser desorption ionization time-of-flight (MALDI-TOF) mass spectrometry has become a fundamental tool for the identification and anal. of peptides and proteins. MALDI-TOF is well suited for the anal. of complex biol. mixts. because

samples are crystd. onto a solid support that can be washed to remove contaminants and salts prior to laser desorption. A no. of approaches for immobilizing samples onto MALDI targets have been put forth. These include the use of different chem. matrixes and the immobilization of samples onto different solid supports. In large part though, the prepn. of MALDI targets has been an empirical exercise that often requires a unique series of conditions for every sample. Here, a simple method for the application of peptide mixts. onto MALDI targets is put forth. This method differs because peptides are added directly to a sample of nitrocellulose dissolved in acetone, allowing them to interact in soln.-phase org. solvent. This soln.-phase mixt. is then spotted to the MALDI target and evapd., forming a homogeneous solid surface for laser desorption. This procedure is robust, highly sensitive, tolerant to detergents, and easily learned. In our hands, the method provides as much as a 10-fold enhancement to the detection of tryptic peptide fragments derived from in-gel digests. (c) 2000 Academic Press.

REFERENCE COUNT: 25

L10 ANSWER 5 OF 9 CAPLUS COPYRIGHT 2003 ACS

ACCESSION NUMBER: 1999:723216 CAPLUS

DOCUMENT NUMBER: 131:334352

TITLE: "Infrared matrix-assisted laser desorption/ionization mass spectrometric analysis of biomacromolecules"

INVENTOR(S): *Hillenkamp, Franz; Koster, Hubert*

PATENT ASSIGNEE(S): Sequenom, Inc., USA

SOURCE: PCT Int. Appl., 205 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent

PATENT NO. KIND DATE APPLICATION NO. DATE

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WO 9957318 A2 19991111 WO 1999-US10251 19990507

WO 9957318 A3 20000323

US 2001055811 A1 20011227 US 1998-74936 19980507

CA 2328110 AA 19991111 CA 1999-2328110 19990507

AU 9938967 A1 19991123 AU 1999-38967 19990507

**EP 1075545** A2 20010214 EP 1999-921861 19990507

DE 19983215 T 20010517 DE 1999-19983215 19990507

JP 2002513917 T2 20020514 JP 2000-547269 19990507

NO 2000005539 A 20010105 NO 2000-5539 20001102

PRIORITY APPLN. INFO.: US 1998-74936 A 19980507

WO 1999-US10251 W 19990507

AB Mixts. contg. a biol. macromol., such as a nucleic acid mol. or a polypeptide, and a liq. matrix, which absorbs IR (IR) radiation, are provided. These mixts. are useful for anal. of the biol. macromol. by IR matrix assisted laser desorption/ionization (IR-MALDI) mass spectrometry. Also provided are processes for analyzing a biol. macromol. using IR-MALDI mass spectrometry. For example, processes for detecting the presence or identity of a biol. macromol. in a sample, or for sequencing a biol. macromol. are provided. IR-MALDI of DNA (700 kDa being the

upper mass limit with current conditions), RNA, IgG, myoglobin, and gramicidin-S-synthetase were demonstrated.

L10 ANSWER 6 OF 9 CAPLUS COPYRIGHT 2003 ACS

ACCESSION NUMBER: 1999:246114 CAPLUS

DOCUMENT NUMBER: 131:11073

TITLE: p-nitroaniline/glycerol: a binary liquid matrix for matrix-assisted laser desorption/ionization analysis

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AB Liq. matrixes for matrix-assisted laser desorption/ionization (MALDI) anal. offer advantages over solid matrixes including increased shot-to-shot reproducibility and the ability to provide conditions more suitable for the retention of noncovalent complexes. Unfortunately, most of the liq. matrixes available do not absorb at UV wavelengths. A 2nd component to increase chromophoric absorption and to permit control over the chem. properties of the matrix can aid in overcoming some of the limitations currently faced by the MALDI technique. In the work reported here, a binary matrix that combines the advantages of the fluidity and low vapor pressure of glycerol with the weak basicity and chromophoric properties of p-nitroaniline is evaluated. Both pos.-ion and neg.-ion spectra were obtained for proteins, carbohydrates and oligonucleotides. The potential for anal. of chelates and noncovalent interactions using this liq. binary matrix is also explored.

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